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PERFORMANCE OF A MASS SPECTROMETRIC MODULATED BEAM DETECTOR FOR GAS-SURFACE INTERACTION MEASUREMENTS

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ARO, Inc.

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FOREWORD

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This technical report has been reviewed and is approved.

Harry R. McDaniel	Edward R. Feicht
2nd Lieutenant, USAF	Colonel, USAF
Directorate of Plans and Technology	Director of Plans and Technology

ABSTRACT

This report describes the performance of an instrument designed to detect molecular flow rates in the presence of the relatively high background gas noise of a space environment chamber. The technique of molecular beam modulation employed consists of three primary elements: (1) the mechanical beam modulator, (2) a quadrupole mass spectrometer, and (3) a lock-in amplifier. Molecular beam intensities of about 1×10^{10} molecules/sec-cm^2 are distinguishable from noise at a background pressure of 2×10^{-7} torr, which corresponds to about 1×10^{14} molecules/sec-cm^2. The detector performance for molecular beams of He, A, N_2, and CO_2 is reported. This high sensitivity makes possible the use of molecular beam techniques for direct measurements of gas condensation rates.

CONTENTS

ILLUSTRATIONS

NOMENCLATURE

A_s	Source orifice area in an oven beam generator, cm^2
C	Capture coefficient, nondimensional
I_o	Oven beam intensity, molecules/sec-cm^2 of collimator area
I_{rc}	Reflected beam intensity entering detector from surface cooled to desired testing temperature, molecules/sec-cm^2 of detector entrance area

I_{rw}	Reflected beam intensity entering detector gage from surface sufficiently warm to prevent beam gas condensation, molecules/sec-cm^2 of detector entrance area
k	Boltzmann constant, ergs/°K
ℓ_{sc}	Source orifice to collimator distance in oven beam generator, cm
m	Molecular mass, gm/molecule
N	Electrical noise at detector output, μv
n_o	Gas density in oven beam generator source plenum, molecules/cm^3
P_{bg}	Background gas pressure in test chamber, torr
S	Electrical signal at detector output, μv
T_o	Source gas temperature, °K
$\bar{v}_i$	Mean molecular velocity entering the ionization region of the detector, cm/sec
$\bar{v}_o$	Mean molecular velocity in the source gas plenum of oven beam generator, cm/sec

SECTION I
INTRODUCTION

At the Arnold Engineering Development Center (AEDC) an aerodynamic molecular beam is being used in the study of interactions between neutral gas molecules and solid surfaces. Of particular interest is the gas-surface interaction involving condensation of the test gas at the surface interface. Such studies may have practical value in the performance evaluation of cryogenic pumping surfaces. In addition, these studies should contribute fundamental information on the condensation process which could aid in the theoretical description of the governing gas-surface interaction mechanism. The AEDC aerodynamic molecular beam generator as described in Ref. 1 was designed specifically for these studies and is capable of producing intense molecular beams on the order of 10^{17} molecules/sec-cm^2 at the target surface.

The nature of the gas-surface interaction experiments is depicted in Fig. 1. A molecular beam of the test gas is scattered by the target

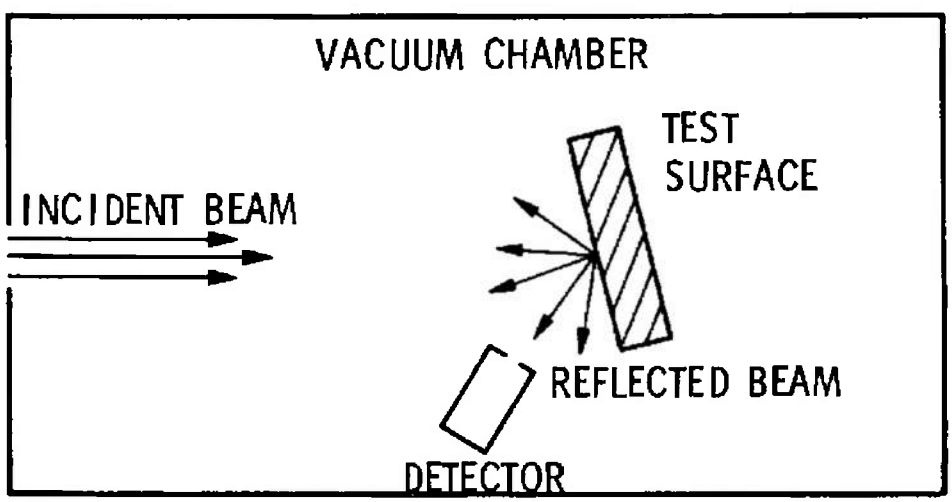

Fig. 1 Molecular Beam Scattering Experiment

surface, and a portion of the reflected beam is intercepted by the detector which is designed to respond to beam intensity (molecules/sec-cm^2 of detector entrance area). The detector is movable in two dimensions to allow scanning throughout the spatial distribution pattern of the reflected gas beam. For the condensation studies, the capture coefficient for the interaction can be measured using the relation:

$$C = 1 - \frac{I_{rc}}{I_{rw}} \tag{1}$$

I_{rc} is the reflected beam intensity entering the detector when the surface is cooled to some desired testing temperature, and I_{rw} is the reflected

beam intensity when the surface is sufficiently warm to prevent condensation. The reflected beam intensity is taken as the difference between the intensities entering the detector when the beam is cycled on and off. The capture coefficient is then the fraction of the incident beam molecules which condense on the surface at the testing temperature.

In principle, a direct measurement and simple calculation of capture coefficient as defined by Eq. (1) is possible. However, for a high value of the capture coefficient the magnitude of the reflected beam intensity entering the detector will be much less than the intensity of the background gas molecules randomly striking the detector entrance. For example, an incident beam of 10^{15} molecules/sec-cm^2 and a capture coefficient of 0.99 will result in 10^{10} molecules/sec-cm^2 entering the detector for a typical detector solid angle. At a test chamber pressure of 10^{-8} torr, the background gas intensity on the detector is about 10^{12} molecules/sec-cm^2. Consequently, a detector capable of extracting the relatively weak reflected beam signal from the stronger background gas signal must be used. The purpose of this report is to describe the design and performance of a molecular beam detector which satisfies this signal recovery requirement for capture coefficient measurements.

SECTION II
DESCRIPTION OF DETECTOR AND EXPERIMENTAL APPARATUS

The widespread use of molecular beams for atomic and molecular interaction experiments has resulted in the development of numerous beam detection techniques. A general review of molecular beam experiments and detectors is given in Ref. 2. One technique which aids in the discrimination between the beam signal and background noise is to modulate the molecular beam at a convenient frequency and then by a-c amplification measure only that part of the signal which has the proper frequency and phase. This is generally referred to as modulated beam or "lock-in" detection, and the technique is described in detail in Refs. 3 and 4. The detector described in this report combines the beam modulation technique with a quadrupole mass spectrometer as the electrical signal source. The use of a mass spectrometer further increases the signal-noise ratio of the detector by rejecting all signals at mass numbers other than the beam mass number. The detectors described in Refs. 3 and 4 are specially fabricated instruments designed to meet the specific requirements of the user, whereas the detection system reported on herein was assembled from commercially available components.

In Fig. 2 the primary components of the detector are shown mounted in the test chamber end of a small oven beam generator. This beam generator produces a molecular beam of known intensity which can be used for detector calibration. The test gas originates in a small gas plenum (or oven) at a sufficiently low pressure to ensure random, free-molecular flow through a thin-walled orifice into a surrounding vacuum. A beam is formed by those molecules passing through a collimating orifice located in the dividing wall between the two vacuum chambers. The resulting beam intensity can then be calculated from kinetic theory as discussed in Ref. 5:

$$I_o = \frac{n_o \bar{v}_o A_s}{4\pi b^2_{sc}} \tag{2}$$

For calibration purposes, the electrical output of the detector can then be compared to the calculated beam intensity.

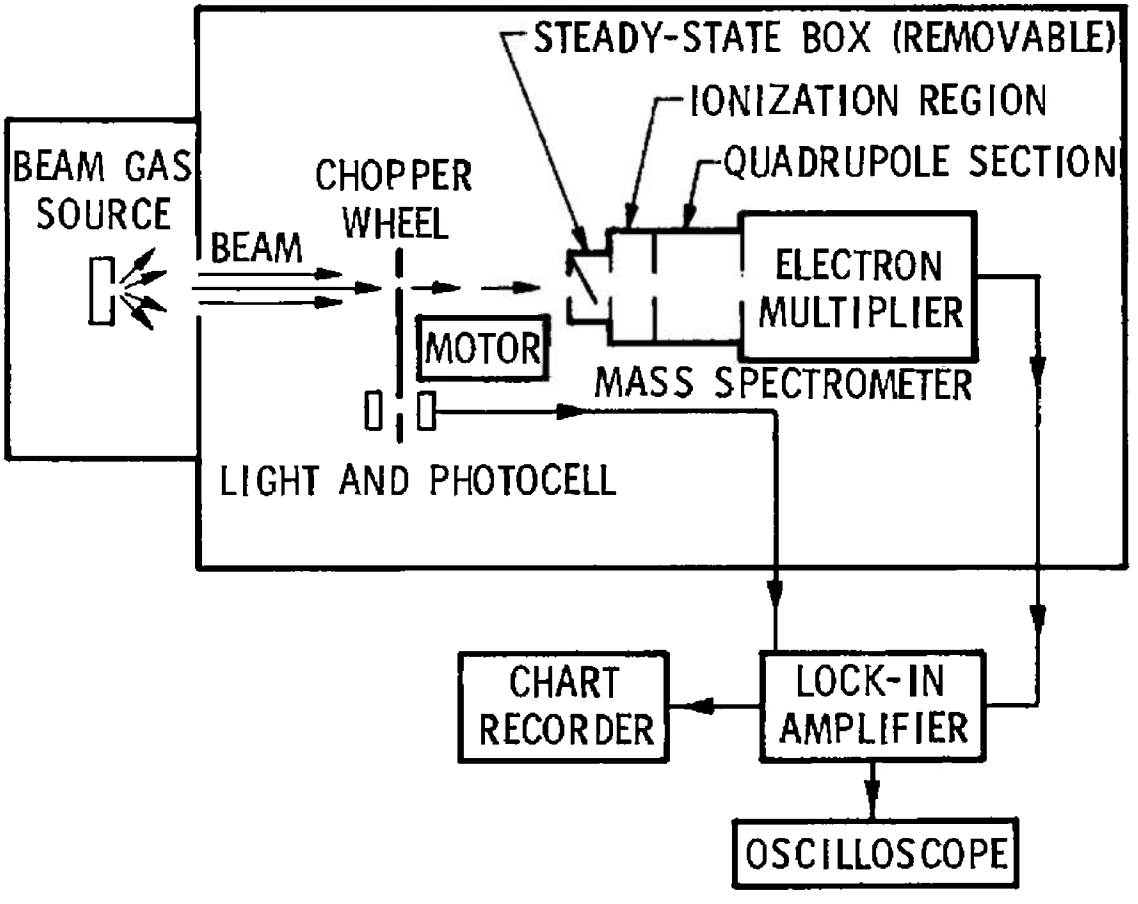

Fig. 2 Schematic of Mass Spectrometric Modulated Beam Detector

As depicted in Fig. 2 the calibrating beam to be detected is chopped into pulses by two slots in a rotating wheel. These slots are 90 deg sectors cut out of a 2-in.-diam aluminum disc. The beam chopper is driven by a small, two-phase, synchronous vacuum motor[1] generally turning at 50 cps. These beam pulses then pass into the ionization region of a quadrupole mass spectrometer[2] which is tuned to the mass number of the beam molecules. The resulting ion current is amplified by an electron multiplier which sends a pulsed electrical signal to the "lock-in" amplifier.[3] This amplifier operates as a narrow band pass amplifier, the center frequency of which is locked to a particular frequency at which the signal has been made to appear. A light and photocell mounted at the chopper wheel transmit to the amplifier the chopping frequency and any variations in chopping frequency. The amplifier increases the signal-noise ratio by amplifying only the electrical signal at the proper frequency and phase with respect to the reference signal from the photocell. The pulsed signal is rectified and integrated into a d-c signal at the output of the "lock-in" amplifier. This signal is read out on a strip chart recorder, and an oscilloscope is used to monitor the a-c tuning of the amplifier.

SECTION III
DETECTOR PERFORMANCE EXPERIMENTS

3.1 OPERATING MODES

The detector has been designed for two modes of operation: (1) as a molecular flow rate detector and (2) as a flow density detector. The mode of operation is determined by the manner in which the beam gas is introduced into the detector ionization region as depicted in Fig. 3. As a molecular flow rate (beam intensity) detector, the beam must pass through the "steady-state" box before entering the ionization region of the mass spectrometer. Since the beam molecules are thereby brought to equilibrium with the walls and baffle of this box, the detector responds only to changes in molecular flow rate. This is the measurement required for capture coefficient determination, and this is the mode of operation for the detector evaluation experiments reported herein. The

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[1]A Globe Industries, Inc. No. 53A416-2.

[2]An EAI Quad 200.

[3]A Princeton Applied Research Model HR-8

box is removable to permit use of the instrument as a flow density detector. In this configuration, the beam pulses pass directly into the ionization region without striking any solid surface. Hence, either changes in flow rate or flow velocity change the density of the beam gas in the ionization region.

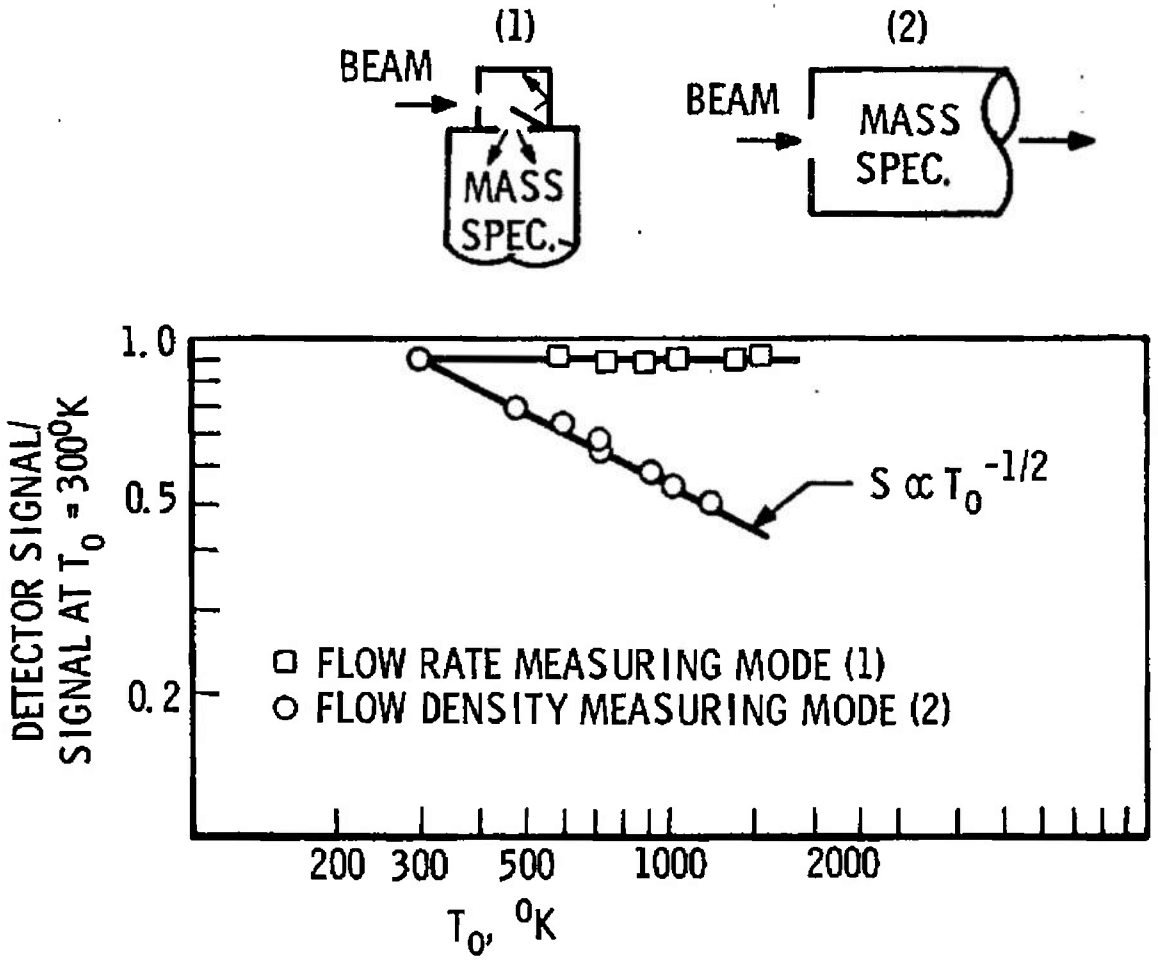

Fig. 3 Detector Response for Two Operating Modes

It has been experimentally verified that the detector output signals are proportional to the desired parameter in the two modes of operation. This was done by recording the detector output signals as a function of molecular beam velocity while maintaining a constant molecular flow rate. By heating the beam gas in the source plenum, the mean molecular velocity is varied as related by the kinetic theory relation:

$$\bar{v}_o = \left(\frac{8kT_o}{\pi m}\right)^{1/2} \tag{3}$$

The detector output signals which are proportional to the gas density in the ionization region can be related to the beam intensity and flow velocity:

$$S \propto \frac{I_o}{\bar{v}_i} \tag{4}$$

In the molecular flow rate mode of operation, the gas velocity entering the ionization region should remain constant as the beam velocity is varied if the "steady-state" box serves to bring the beam gas to the same equilibrium state. In this condition $\bar{v}_i$ will be constant. The resulting detector signals should then remain constant as the beam velocity is varied and the beam intensity held constant. In the flow density mode of operation the molecular beam passes directly through the ionization region without striking any solid surfaces. As related by Eq. (4), the detector signals should then vary inversely with the beam velocity, which in this case is also the gas velocity entering the ionization region.

In Fig. 3 the detector output signals for the two modes of operation are shown plotted against source gas temperature which controls the beam velocity, Eq. (3). For the data taken in the flow rate mode of operation, the detector signals remain essentially constant as the temperature increases, and this indicates proper performance of the "steady-state" box. The data taken for the flow density mode of operation indicate that the detector signal response is in accordance with Eqs. (3) and (4) since the straight line through these points represents a square-root relation on a log-log plot.

3.2 SIGNAL-NOISE PERFORMANCE

The performance of the detector has been experimentally determined by recording the output of the lock-in amplifier as a function of beam intensity. By varying the pressure in the beam gas source chamber, the beam intensity could be varied over a wide range. As long as this source pressure was sufficiently low to ensure free-molecular flow through the source orifice, the resulting beam intensity could be calculated by using Eq. (2). For a given beam intensity, the detector output signal was recorded; and then with the beam turned off, the noise signal remaining at the detector output was also recorded. The magnitude of the detector noise signal was arbitrarily taken as one-half the value of the average peak-to-peak oscillations on the chart recorder as shown in Fig. 4. This is also considered the minimum magnitude of a beam-on signal which could be distinguished from the noise signal - i.e., a signal equal to noise condition. A small aluminum flag was located at the beam

collimating orifice which could be remotely positioned to cover the collimator, and hence to turn the beam on and off. In this manner the signal-noise response of the detector has been determined for four different beam gases: He, A, N_2, and CO_2. Two typical data points are shown in Fig. 4. The upper data point represents a signal-noise ratio of about 10, and the lower represents a ratio of about 3. As the signal-noise ratio decreases, it is evident that a minimum detectable signal would occur for a signal-noise ratio of unity.

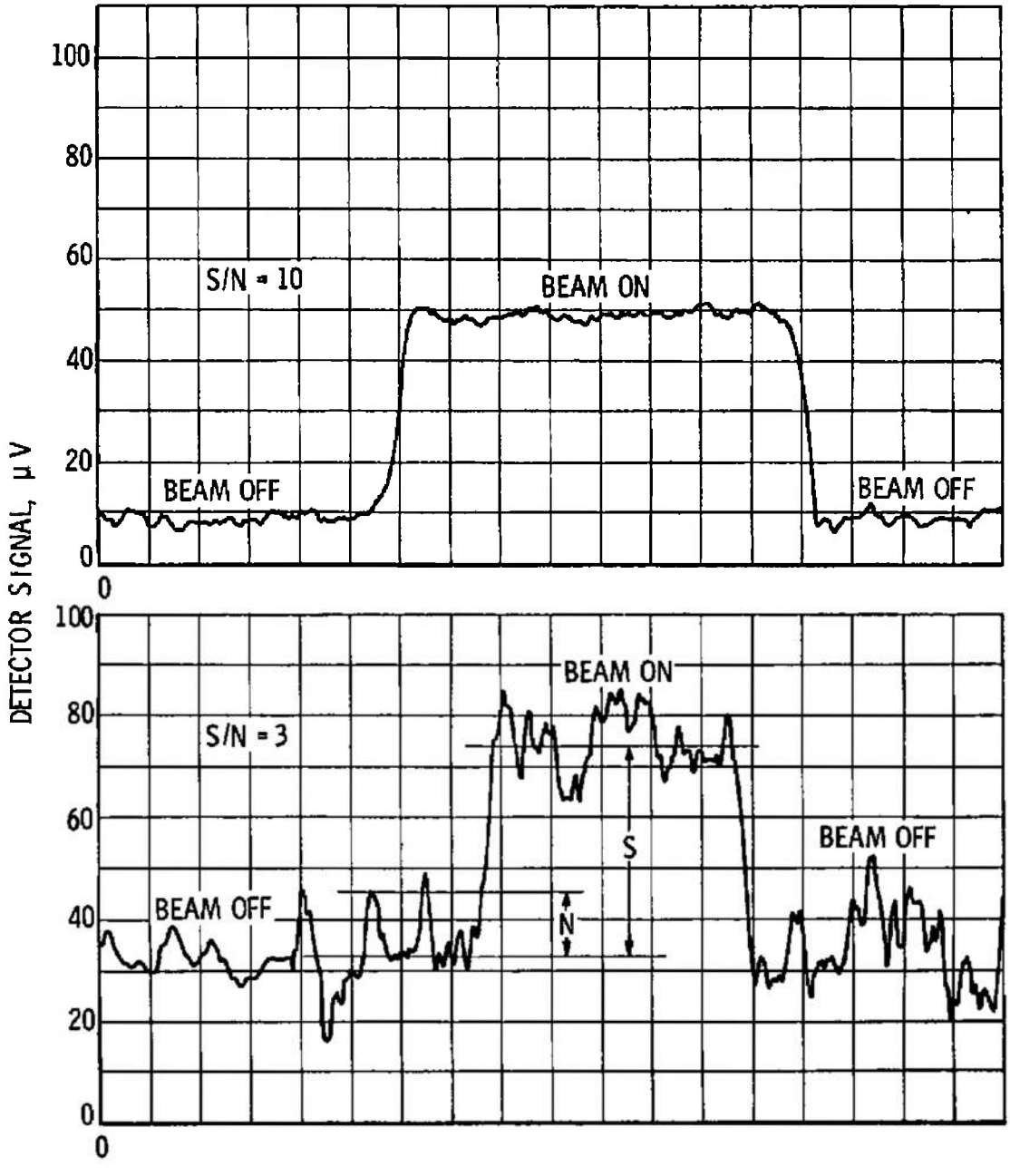

Fig. 4 Detector Response for Two Different Signal-Noise Ratios

The results of these experiments are shown in Fig. 5. Beam intensities have been computed using Eq. (2). A horizontal dashed line has been drawn to represent the intensity level of the background gas at the test chamber pressure, which was 2×10^{-7} torr. The background intensity level of the beam gas mass number is indicated on each curve. There are two important detector characteristics indicated in this figure. First, since all curves are straight lines with unity slope on a log-log scale, the detector signal varies in a linear manner to changes in beam intensity. Secondly, beam intensities well below the level of the background gas intensity can be detected. Experimental data are shown down to about 10^{12} molecules/sec-cm^2 in a background of 10^{14} molecules/sec-cm^2.

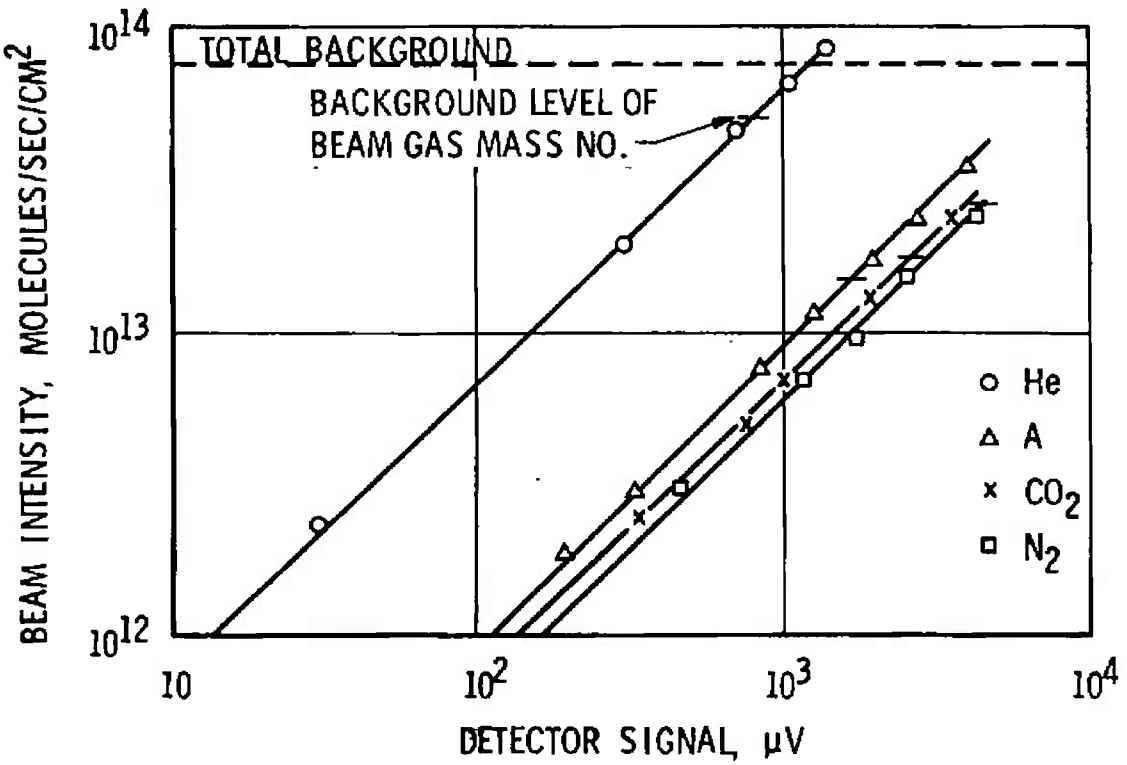

Fig. 5 Beam Detector Performance

As previously stated, the noise remaining at the detector output with the beam turned off was also recorded. By using the signals produced by beam intensities of 1×10^{12} molecules/sec-cm^2 as a standard beam condition, a signal-noise ratio can be written:

$$\left(\frac{S}{N}\right)_s = \frac{\text{Signal produced by standard beam}}{\text{Detector noise signal}} \qquad (3)$$

This ratio can be used as a criterion for determining a minimum detectable beam intensity for a particular background noise condition:

$$I_{min} = \frac{\text{Standard beam intensity}}{(S/N)_s} \tag{4}$$

This is the beam intensity which would produce a detector signal equal to the detector noise signal. The values of $(S/N)_S$ together with the computed minimum detectable beam intensities are listed in the following table. Also shown are the values of electrical sensitivity computed from the currents generated by the electron multiplier.

Gas	He	A	CO_2	N_2
SENSITIVITY, $\frac{\text{amp}}{\text{molecules/sec-cm}^2}$	1.61×10^{-23}	1.11×10^{-22}	1.45×10^{-22}	1.67×10^{-22}
S/N at 2×10^{-7} torr for 1×10^{12} molecules/sec-cm^2	2	26	24	30
MIN. DETECTABLE INTENSITY at 2×10^{-7} torr	5×10^{11}	3.8×10^{10}	4.2×10^{10}	3.3×10^{10}
MIN. DETECTABLE INTENSITY at System Limiting Noise	2.3×10^{10}	1.7×10^{9}	1.5×10^{9}	1.2×10^{9}

The detector noise data from the experiments represented in Fig. 4, are for one background chamber pressure condition - 2×10^{-7} torr. If the background pressure were lowered, it would be expected that the detector noise would be lowered, which would reduce the value of the minimum detectable beam intensity. The manner in which detector noise varied as a function of background gas pressure was determined by bleeding in CO_2 gas to the test chamber while the detector was tuned to the CO_2 mass number. The results of these experiments are shown in Fig. 6. As the background pressure was reduced, the detector noise decreased. The straight line drawn in Fig. 5 represents a square-root relation on a log-log scale. Note that the variation of detector noise signal with the square root of the background gas density is in agreement with the "shot noise" theory of statistical fluctuations. As described in Ref. 6, the "shot noise" arising from random ion emission is inversely proportional to the square root of the molecular density in the detector ionization region. A horizontal line has been drawn at the value of system electronic noise remaining when no CO_2 ions are being transmitted through the quadrupole mass spectrometer. This represents the lower limit for noise reduction which can be achieved by decreasing the background pressure. Using this lower limit for noise, a minimum detectable beam intensity can be calculated

by using Eqs. (3) and (4), as before. These values, shown in the last row of the preceding table, represent a practical lower limit of detector operation.

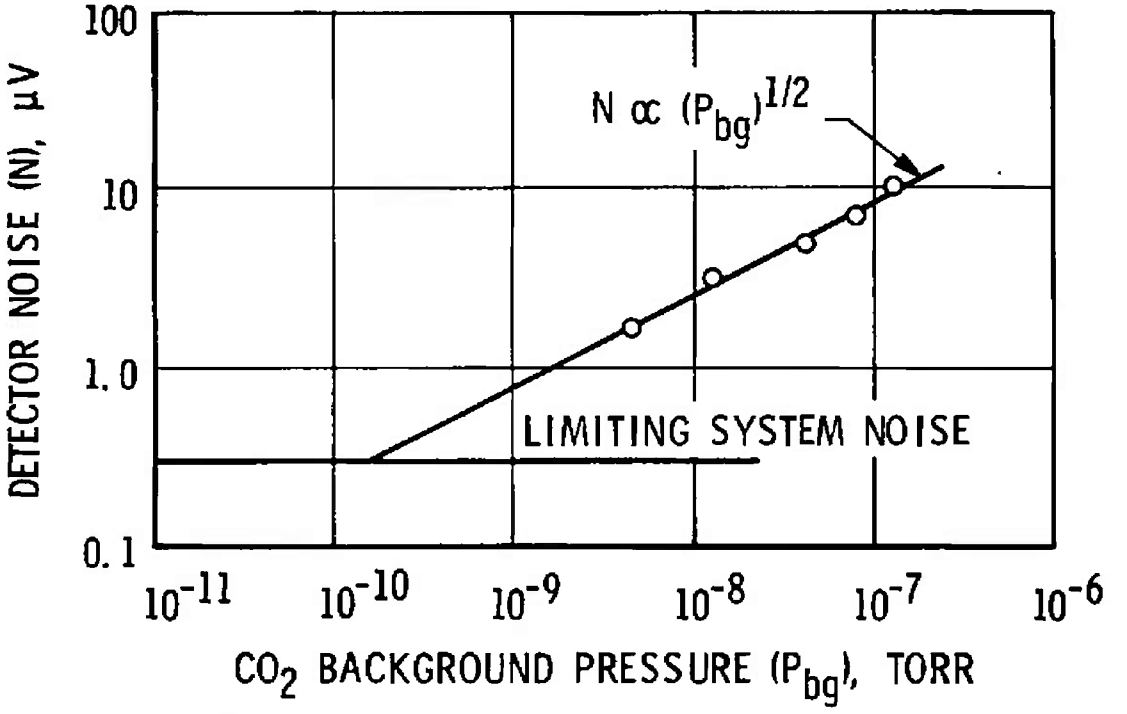

Fig. 6 Effect of Background Pressure on Detector Noise

The signal-noise ratio was also determined as a function of chopping frequency between 20 and 200 cps and as a function of the mass spectrometer control settings of emission current, electron multiplier voltage, and resolution. None of these variables produced any significant change in signal-noise ratio. Any change which produced an increase in signal also produced a similar increase in noise.

SECTION IV
CONCLUSIONS

A mass spectrometric modulated beam detector has been developed from commercially available components which satisfies the requirements of gas-surface interaction measurements of condensation rates. The detector is capable of recovering signals from beam intensities three orders of magnitude below the level of the total background gas intensity at 2×10^{-7} torr.

REFERENCES

1. Brown, R. F. and Heald, J. H. "Description and Performance of a Molecular Beam Chamber Used for Cryopumping and Adsorption Pumping Studies." AEDC-TR-66-135 (AD 641388), October 1966.

2. Trischka, J. W. Methods of Experimental Physics 3: Molecular Physics. Edited by D. Williams, Academic Press, New York, N. Y., 1962

3. Fite, W. L. and Brackmann, R. T. "Collisions of Electrons with Hydrogen Atoms. I. Ionization." Phys. Rev., Vol. 112, No. 4, 1958, pp. 1141-1151.

4. Fite, W. L. and Datz, S. "Chemical Research with Molecular Beams." Annual Review of Physical Chemistry, Annual Reviews, Inc., Stanford, California, Vol. 14, 1963, pp. 61-68.

5. Ramsey, N. F. Molecular Beams. Clarendon Press, London, 1956.

6. Bennewitz, H. G. and Wedemeyer, R. "Molecular Beam Detector with Electron Bombardment Ionization and Four-Pole Mass Filter." Z. Physik, Vol. 172, No. 1, 1963, pp. 1-18.

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